FABRICATION AND CHARACTERIZATION OF SILICON NITRIDE THIN FILM PLANAR WAVEGUIDES PRODUCED BY RF MAGNETRON SPUTTERING TECHNIQUE

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DEDICATION

To my parents, my brothers, my sister, my wife and my son
Without whom none of my success would be possible.

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ABSTRACT

Silicon nitride based planar waveguides play an important role in biosensing applications. Conventional fabrication of silicon nitride waveguides utilizes the chemical vapor deposition (CVD) technologies. Silicon nitride waveguide fabrication through magnetron sputtering is still unexplored and a few literatures are available only on microelectronic and optoelectronic applications. In current study, the silicon nitride thin film planar waveguides were fabricated on SiO_2 coated silicon substrates by RF magnetron sputtering technique. Sputtering power, sputtering pressure and target to substrate distance was varied from 100 to 300 W, 5 to 15 mTorr and 8 to 14 cm respectively to investigate the influence of sputtering parameters on film properties. The deposited films were characterized with FESEM, AFM and surface profile to observe the surface morphology. The structure and composition of deposited thin films were characterized with XRD and EDX spectroscopy techniques respectively. Optical properties such as refractive index and optical transmission were investigated through spectral reflectance and UV-VIS spectroscopy techniques respectively. After a detailed analysis, suitable sputtering parameters were selected as sputtering power 300 W, sputtering pressure 5 mTorr and target to substrate distance 14 cm to fabricate the asymmetric planar waveguide. The purpose of this fabrication was to investigate the optical propagation and to measure the propagation loss using prism coupling technique. The optical properties and thickness was determined first using spectroscopic ellipsometry technique. The thickness of waveguide was intended to greater than 400 nm as a requirement of prism coupling technique. The fabricated waveguide demonstrated a successful propagation of light at wavelength, λ = 448 nm and 633 nm. The estimated loss at 633 nm was 20 – 25 db/cm. This study supports the possibility of producing silicon nitride thin film planar waveguides by RF magnetron sputtering technique

ABSTRAK

Pemandu rambatan gelombang berasaskan silicon nitrid memainkan peranan penting dalam aplikasi pengesan bio. Kaedah konvensional adalah menggunakan teknologi wap kimia (CVD). Fabrikasi pemandu pemandu rambatan gelombang menggunakan magnetron sputtering masih kurang dikaji dan hanya terdapat beberapa rujukan dalam aplikasi mikro dan optoelectronik. Kajian ini menumpukan kepada fabrikasi pemandu rambatan gelombang planar silikon nitrid di atas subtract silikon oksida menggunakan teknin “RF magnetron sputtering”. Parameter seperti kuasa, tekanan dan jarak antara target ke substrat diubah antara 100 – 300 W, 5 – 15 mTorr dan 8 – 14 cm setiap satu. Filem nipis yang didepositkan dianalisa menggunakan FESEM, AFM dan profiler untuk mengkaji morfologi permukaan. Struktur dan komposisi filem nipis yang telah didepositkan dianalisa menggunakan Teknik XRD dan spektroskopi EDX. Ciri-ciri optik seperti indeks biasan dan transmisi optik telah dianalisa melalui pantulan spektrum dan teknik spektroskopi UV-VIS. Setelah analisis yang terperinci, parameter “sputtering” yang sesuai adalah kuasa sputtering 300 W. Tekanan “sputtering” sebanyak 5 mTorr dan jarak antara sasaran dan substrat adalah 14 cm untuk fabrikasi simetri pemandu rambatan gelombang. Tujuan fabrikasi adalah untuk menyiasat perambatan optik dan untuk mengukur atinuasi gelombang menggunakan teknik “prism coupling”. Ciri-ciri dan ketepalan optik telah ditentukan melalui teknik spektroskopi ellipsometri. Ketebalan pemandu rambatan gelombang melebihi 400 nm sebagai memenuhi kepertuan teknik “prism coupling”. Pemandu rambatan gelombang yang telah difabrikasi menunjukkan kejayaan perambatan cahaya pada gelombang 448 nm dan 633 nm. Anggaran atinuasi gelombang pada 633 nm adalah 20 – 25 db/cm. Kajian ini disokong kemungkinan mengeluarkan pemandu rambatan gelombang planar silikon nitrid oleh teknik “RF magnetron sputtering”.

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LIST OF SYMBOLS AND ABBREVIATIONS

d_c	- Collision diameter
e	- Charge of electron
$\boldsymbol{E}$	- Electric field vector
$\boldsymbol{H}$	- Magnetic field vector
h	- Thickness of core
i_s	- Current density
k	- Wave vector
k	- Extinction co-efficient
m	- Mode of propagation
n_c	- Refractive index of upper cladding
n_f	- Refractive index of core
n_s	- Refractive index of lower cladding
r	- Radius vector
v	- Velocity of light in vacuum
v_e	- Velocity of electron
α	- absorption co-efficient
β	- Propagation constant
ω	- Frequency of light
ω_c	- Cutoff frequency
λ_o	- Wavelength of light in vacuum
λ_c	- Cutoff wavelength
λ_{mfp}	- Mean free path
φ	- Angle of light ray w.r.t normal to interface plane
Δn	- Refractive index difference between core and cladding
AFM	- Atomic Force Microscopy
AGZO	- Aluminum Gallium Zinc Oxide
AZO	- Aluminum doped Zinc Oxide

B	- Magnetic field
C	- Capacitance
CVD	- Chemical Vapor Deposition
CRT	- Cathode Ray Tube
D	- Dimension
DC	- Direct Current
DI	- Deionized Water
E_1	- Energy of incident ion
E_b	- Surface binding energy
EDX	- Energy Dispersive X-ray
FESEM	- Field Emission Scanning Electron Microscopy
GM/V	- Gas Main Valve
HCG	- Human Chorionic Gonadotropin
IGZO	- Indium Gallium Zinc Oxide
IPA	- Iso Propyle Alcohol
ITO	- Indium Tin Oxide
ITiO	- Indium doped Titanium Oxide
LPCVD	- Low Pressure Chemical Vapor Deposition
M_1	- Mass of incident ion
M_2	- Mass of target atom
MiNT-SRC	- Micro and Nano Technology Shamsuddin Research Center
N_A	- Avogadro’s number
NIR	- Near Infra Red
ONO	- Oxide Nitride Oxide
P	- Pressure
PECVD	- Plasma Enhanced Chemical Vapor Deposition
PVD	- Physical Vapor Deposition
R	- Ideal gas consyant
RF	- Radio Frequency
SCCM	- Standard Cubic Centi Meter
S	- Sputtering yeild
SR	- Spectral Reflectance
STP	- Standard Temperature Pressure
T	- Transmission

TE	- Transverse Electric
TM	- Transverse Magnetic
TMP	- Turbo Molecular Pump
TIR	- Total Internal Reflection
T/V	- Throttle Valve
UTHM	- University Tun Hussein Onn Malaysia
UV-VIS	- Ultra Violet – Visible
W	- Watt
XRD	- X-Ray Diffraction

LIST OF PUBLICATIONS

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CHAPTER 1

INTRODUCTION

1.1 Introduction

The propagation of electromagnetic radiations in a homogeneous media has been a subject of interest because of its wide range applications. The phenomenon is described as electromagnetic wave guiding and the media is called waveguide. Planar or slab waveguide is the simplest structure that supports electromagnetic wave guiding. It is a multilayer structure in which a high refractive index media is sandwiched between low refractive media in planar geometry. An illustration of symmetric planar waveguide is shown in Figure 1.1.

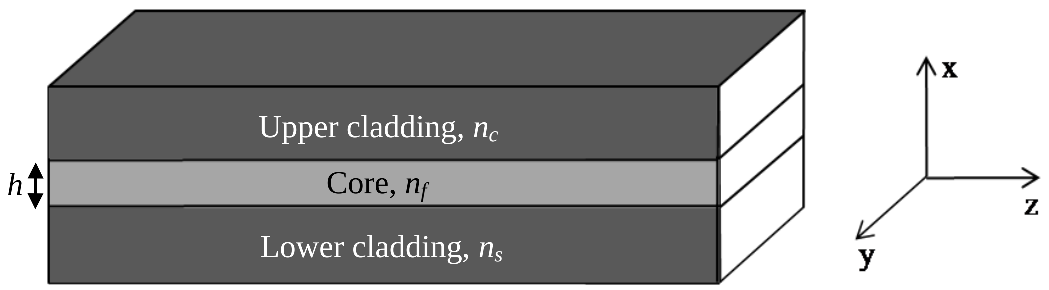

Figure 1.1: Illustration of symmetric planar waveguide

According to Figure 1.1, n_s, n_f and n_c are the refractive indices of lower cladding, core and upper cladding respectively such that $n_f > n_s \geq n_c$ and 'h' is the thickness of core layer. The light beam is coupled with the angle larger than the critical angle, it is confined in the core layer due to total internal reflection (TIR) from the interface.

Planar optical waveguides based sensing devices with high sensitivity, selectivity and rapid detection have already been demonstrated for clinical, environmental, industrial and military applications (Duval *et al.*, 2012; Long *et al.*, 2013). The sensitivity of planar waveguides has a direct relation with the refractive index of core layer. Due to this, the high refractive index materials such as TiO_2, Ta_2O_5, Nb_2O_5 and Si_3N_4 have attracted the researcher's attention because of their low propagation losses and high stability (Aquino *et al.*, 2013; Bauters *et al.*, 2011; Bradley *et al.*, 2010; Muttalib *et al.*,2014).

Silicon nitride is one of the promising ceramic materials that was developed in 1960`s and 70`s in search of dense, highly stable and tough materials. The remarkable chemical, mechanical and electronic properties of silicon nitride widen it scope to numerous applications such as anti reflective coatings, dynamic random access memory, gate dielectrics in field effect transistors, protective coatings and waveguide devices (Signore *et al.*, 2012). The properties such as high and tunable refractive index, known surface chemistry and low propagation losses fascinated researchers to exploit these films for development of highly sensitive waveguide based devices for sensing purposes.

Waveguide fabrication is the most crucial step in developing an effective device. Fabrication techniques can be categorized as chemical vapor deposition (CVD) and physical vapor deposition (PVD). The deposition of thin films using CVD techniques is done by exposing the substrates to one or more volatile precursors which either react or decompose on the substrate surface. In PVD techniques, physical processes such as high temperature vacuum evaporation with subsequent condensation or plasma sputter bombardment are exploited to deposit the required thin films.

Magnetron sputtering is one of the most common PVD process used to deposit high quality and uniform thin films. The process involves the bombardment of high energy plasma ion on the surface of sputtering target, which causes to dislodge a physical target atom, vaporize and then condense onto substrate to form a thin film (Chapman, 1980). Magnetron sputtering deposition with high density, good deposition rate and flat surface at low temperatures has already been published in literatures (de Castro *et al.*, 2012; Yuste *et al.*, 2012). Sputtering parameters have significant influences on the deposited thin film properties.

Preparation of silicon nitride films by other methods such as low pressure chemical vapor deposition (LPCVD), sol gel, thermal evaporation, plasma enhanced chemical vapor deposition (PECVD), ion plating and magnetron sputtering also has been reported. (Ali, Khan, & MatJafri, 2015; Karouta *et al.*, 2012; Morin *et al.*, 2012). The films deposited by magnetron sputtering show a delicate control on film properties with variation in deposition parameters. The properties such as growth rate, refractive index, surface roughness, film homogeneity, surface density and chemical composition have imperative effect in development of planar waveguides. Precise control gives the opportunity for optimization of deposition parameters to produce low loss planar optical waveguides.

1.2 Background of study

Magnetron sputtering is one of the PVD process to deposit thin films of virtually all materials due to availability of sputtering targets commercially. Silicon nitride thin films are under study due to its remarkable structural, electrical and optical properties which could be use for several applications.

Planar waveguide is a multiple layered structure used to confine light and pass over the required distance. The light is incident on a transparent interface separating two mediums of different refractive indices. Light refract through the interface along with partial reflection. Increasing incident angle above the critical angle gives the complete reflection from interface, name as total internal reflection (TIR). The wave guiding phenomenon is based on TIR for guiding the light over a required path.

During TIR phenomenon, a small portion of light still penetrates through interface whose intensity exponentially decayed over some hundred of nanometers. This portion of light which is highly sensitive to the refractive index of interface is called evanescent wave. Waveguides can be used for sensing applications by exploiting evanescent wave as a probe to detect a change in refractive index due to a binding event and later affect the propagation of light in the waveguide (Kozma *et* *al.*, 2014). This probing technique gave rise to several detection protocols such as phase modulation and intensity modulation for sensing application.

Heideman and colleagues first demonstrated the silicon nitride based oxide/nitride/oxide (ONO) waveguide in 1991. The fabrication of ONO waveguide structure was started with growing of silicon dioxide lower cladding by thermal oxidation technique on silicon substrates. A core layer of silicon nitride was deposited through LPCVD technique. The structure was completed by depositing silicon dioxide upper cladding through PECVD technique. They utilized ONO structure for the detection of 2.5 ng/ml of Human Chorionic Gonadotropin (HCG). The detection protocol used for this experiment was phase modulation with a phase shift of 0.6 π (Heideman, Kooyman, & Greve, 1991).

Schipper from the same group fabricated the ONO structure from the same procedure and utilized it to develop a rather simple detection protocol. The ONO structure was partly covered with upper cladding. Any immune reaction occurred in uncovered surface of sensor resulted a change in critical angle. Therefore, the sensor was named as critical sensor (Schipper *et al.*, 1995).

Shirshov with his colleagues developed the ONO structure with same procedure in 1998. They exploited ONO waveguide as planar polarization interferometer. The detection protocol was based on measuring the polarization state of light beam after the interaction with immunoreactions. The sensor was able to detect as low as 10 ng/ml concentration (Shirshov *et al.*, 1998). Nabok's group exploited the ONO waveguide fabricated by the same method for the detection of 10 ppb of imidacloprid pesticide using the intensity modulation detection protocol (Nabok, Haron, & Ray, 2003).

The conventional fabrication of ONO waveguide involves three deposition technologies. The first layer of SiO_2 lower cladding is grown by thermal oxidation on silicon substrates followed by the silicon nitride core layer deposition by LPCVD. Then a protective upper cladding of SiO_2 is deposited by PE CVD technique.

The thermal oxidation technique is a diffusion based process executed at temperature >1000^{o}C using silicon substrates in oxygen ambient. The reaction takes place as presented in Equation 1.1.

$$Si + O_2 \xrightarrow{\text{high temp.}} SiO_2 \qquad (1.1)$$

Hereafter, silicon nitride deposition on SiO_2 substrates is performed by LPCVD technique. Either silane (SiH_4) or dichlorosilane (SiH_2Cl_2) gas is used as silicon precursor with ammonia gas. The reaction takes place at temperature > 600^oC as shown in Equation 1.2.

$$3SiH_4 + 4NH_3 \xrightarrow{\text{high temp.}} Si_3N_4 + 12H_2 \tag{1.2}$$

Further the SiO_2 upper cladding layer is deposited using PECVD technique in silane and nitrogen filled environment. The reaction takes place as shown in Equation 1.3 at temperatures lower than LPCVD due to plasma enhancement.

$$SiH_4 + 2N_2O \xrightarrow{\text{high temp.}} SiO_2 + 2N_2 + 2H_2 \tag{1.3}$$

The waveguide structure showed very low propagation losses. The refractive indices of silicon dioxide upper and lower cladding layer was measured as n = 1.46. The refractive index of silicon nitride core layer was n ~ 2.0. That made the refractive index contrast Δn ~ 0.5. The deposited films are usually high in density. However, despite of producing high quality optical waveguides, the conventional fabrication technology bears some disadvantages as follows: the involvement of toxic gases, incorporation of H_2 gas which causes the degradation of optical and structural properties and elevated temperatures that are inevitable for H_2 removal.

Study shows the silicon nitride deposition by reactive magnetron sputtering technique using Si sputtering target in N_2 ambient (Mousinho *et al.*, 2012). The films were deposited at low temperatures and were free of hydrogen. Therefore sputtering technique has an advantage over current fabrication technology as simple, safe and sequential process. However several sputtering parameters need to optimize in order to produce the good quality waveguide.

A few studies of silicon nitride deposition are reported using stochiometric silicon nitride target. Non reactive approach prevents the incorporation of reactive gas in the process which is good for reasons such as oxidation of target surface, cost effective, minimizing the handling arrangements of extra nitrogen source. Jessica Sandland (Sandland, 2004) deposited the low loss optical waveguides by co-

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